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Layered structures consisting of metals, ceramics and polymers are used extensively for signal and power distribution both at the chip level and in electronic packages. The structural integrity of the interfaces between layers in such structures is an important consideration in the design and fabrication of a variety of devices and components. Various variables including surface composition and structure that control interfacial adhesion are investigated. Interfaces between metal/ceramic, metal/polymer and between polymer/polymer are studied in the research reported.

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Recently it has been recognized that thermal stress-induced damages in metallizations such as hillock and void growth can be an important reliability concern especially at the chip level. The evolution and relaxation of thermal stress, the driving force for such damages, is dependent of the deformation mechanisms in the metallization and the interactions between the metallization and the substrate. The latter leads to the effect of constrained deformation which is of fundamental interest in the study of composite materials systems. Continuous microindentation and indentation load relaxation techniques are developed to probe the effects of constrained deformation in metallizations of technological interests.

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FUNDAMENTAL STUDIES OF ELECTRONIC PACKAGING MATERIALS &

STRUCTURES: SURFACES AND INTERFACES

FINAL REPORT

C. B. CARTER, D. L. KOHLSTEDT, E. J. KRAMER, C-Y. LI, A. L. RUOFF

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STATEMENT OF THE PROBLEM STUDIED

Layered structures consisting of metals, ceramics and polymers are used extensively for signal and power distribution both at the chip level and in electronic packages. The structural integrity of the interfaces between layers in such structures is an important consideration in the design and fabrication of a variety of devices and components. Various variables including surface composition and structure that control interfacial adhesion are investigated. Interfaces between metal/ceramic, metal/polymer and between polymer/polymer are studied in the research reported.

Recently it has been recognized that thermal stress-induced damages in metallizations such as hillock and void growth can be an important reliability concern especially at the chip level. The evolution and relaxation of thermal stress, the driving force for such damages, is dependent of the deformation mechanisms in the metallization and the interactions between the metallization and the substrate. The latter leads to the effect of constrained deformation which is of fundamental interest in the study of composite materials systems. Continuous microindentation and indentation load relaxation techniques are developed to probe the effects of constrained deformation in metallizations of technological interests.

RESEARCH SUMMARY: C. B. CARTER

Surface structures are typically deduced from LEED experiments. LEED has been described as being as important for determining surface structures as x-ray diffraction has been in determining bulk crystal structures. However, LEED experiments are necessarily performed under ultra-high vacuum (UHV) conditions on carefully sputtered and annealed planar specimens. This surface technique can provide structural information from uniform surface terraces and from periodic arrays of defects, such as surface steps, but, the lateral spatial resolution of LEED limits the structural information which can be interpreted from local or aperiodic surface defects such as steps, ledges, facets and kinks. Surface structures on certain ceramic materials have recently been analyzed at very high spatial resolutions with the scanning tunneling microscope (STM) and the atomic-force microscope (AFM). However, at present it is difficult to apply STM to defect structures on the surfaces of wide band gap insulators (e.g., a-Al_2O_3) and AFM has only recently been used to probe the surface structure of BN. In contrast to these scanned-tip microscopies, TEM provides the added benefit of a range of resolutions (from the scale of a few microns to subnanometer) and the same surface defect can be monitored in the TEM during a sequence of thermal and/or chemical processing treatments.

TEM can provide greatly improved lateral resolution of surface topography when compared to LEED. It has been shown that conventional TEM can provide nanometer-scale resolution of surface structures whereas the spatial resolution of LEED has been estimated at ~20nm to 50nm. The technique is perhaps best suited for studying surface defects such as steps, facets and kinks since this surface topography can be resolved from the scale of several microns to the sub-nanometer level. We have now shown that the surface topography of such ceramics as Al_2O_3, SiC and AlN can be studied using a bright-field imaging technique. The thinned TEM sample is heat-treated in a manner which might occur during actual processing. It is then examined in the TEM and the surface topology mapped to determine the extent and characteristics of surface faceting. The next step, which is the emphasis of the present study, is the deposition of metal onto the TEM sample. We have thus shown directly that the topology of the surface directly influences the initial growth of the metal layer. The nuclei form at steps on the surface of the ceramic. Their orientation can be determined by the crystallography of the surface step.

Not only is the lateral resolution of surface topography studies much greater for TEM compared to LEED, but the TEM can be used to study materials in a non-UHV environment. We have extended our study of the surface topography of Al_2O_3, MgO, spinel and SiC to include AlN. The thinned TEM sample is heat-treated in a manner which might occur during actual processing (e.g., in air for the oxides–not UHV). We are heat-treating the AlN samples both in air and in pure nitrogen. The sample is then examined in the TEM and the surface topology mapped to determine the extent and characteristics of surface faceting. Our major advance has been to optimize the conditions for heat-treating AlN.

As a complementary study, we have analyzed a series of alumina samples coated, using the sol-gel process, with doped alumina. This set of samples has been prepared by Prof. Burlitch's group and are similar to those studied by Prof. Kohlstedt using the microindentor technique. The approach of depositing the sol-gel directly onto the TEM sample is still being examined as an alternative to the conventional thick film technique. When this procedure can be perfected, it will provide a new approach for studying sol-gel modification of surfaces.

RESEARCH SUMMARY: D. L. KOHLSTEDT

A systematic series of experiments have been carried out to develop a fundamental understanding of the contribution of chemical bonding to adhesion of metal thin films to ceramic substrates. Focus was given to the Cr-Al_2O3 system. To investigate the effect of chemical bonding, as opposed to mechanical interlocking, the composition of the surfaces of alumina substrates were modified by applying sol-gel coatings of alumina doped with various transition metals, including Cr, Mo, and W (Burlitch et al., 1989) as well as Ni and Ti (DeMott, 1989; Kanai et al., 1990). Chromium was e-beam deposited on pure-alumina and doped-alumina substrates, and continuous microindentation testing was then used to probe the adhesion of the Cr metal to the various Al_2O3 substrates.

To analyze the microhardness versus depth data obtained during indentation of the Cr thin film, a zeroth-order model based on a volume law of mixtures was employed. By fitting the hardness measured for Cr deposited on the Al_2O3 substrates with a weighted average of the hardnesses separately measured on bulk chromium and pure or doped alumina, an adhesion parameter was obtained. Physically, this adhesion parameter can be related to the constraint exerted by the metal-ceramic interface on the plastic flow in the metal thin film. As adhesion (shear strength) of the interface increases, plastic flow in the metal film becomes more difficult; as a result, the adhesion parameter increases in magnitude.

The results of these experiments and the related analysis provided by a marked linear correlation between the increase in adhesion strength and the effective free energy of oxidation of the cations in the ceramic substrate. The effective free energy of oxidation in this context is defined as the weighted average (weighted by mol % of the oxide phases present) of the Gibbs free energy of oxidation of each cation present at the surface of the substrate. Thus, for example, as the Cr:Al ratio increased from 0.02 to 3.0, the adhesion parameter increased from 0.60 to 0.75. When Cr in the Al_2O3 substrate was replaced by Mo or W with the same 3:1 metal to aluminum ratio, the adhesion parameter increased from 0.85 and 0.89, respectively. These changes in composition correspond to increases in the effective free energy of oxide formation from - 375.1 to -281.3 to -190.1 to -189.1 kcal/mol.

Thus, this study has shown very clearly and quantitatively that the adhesion strength of metals to ceramics can be controlled and engineered by careful consideration of the chemical aspects of bonding between the thin film and the substrate materials. In the present case, this chemical effect on bonding occurs because reaction of the chromium with the surface oxide ions results in the formation of metal oxide-oxygen-chromium bonds, at the partial expense of metal-oxygen bonds; if the oxide is forced to take on extra electron density from the chromium (which is partially oxidized in the process of bond formation), it results in partial population of the empty band of the oxide, an unfavorable process. The more readily this electron transfer occurs, however the stronger the Cr-O bond will be. Consequently, availability of low-lying, empty band will facilitate the Cr-O bond formation and the strength of adhesion between the Cr thin film the doped Al_2O3 substrate.

RESEARCH SUMMARY: E. J. KRAMER

The control of cracking along interfaces between a rigid polymer and either another polymer or a non-polymeric material is of interest in a number of critical applications. For example in electronic packaging, polymeric dielectric layers between metals and semiconductors are being used increasingly because of their low dielectric constant. Such packages may eventually consist of many alternating layers. Failure of the interfaces (polymer-polymer, polymer-metal, polymer-semiconductor) is invariably deleterious to the electrical and thermal performance of the package and must be avoided despite imposed stresses due to temperature and humidity cycling.

In his work under this grant, Kramer has shown that he can dope homopolymer layers with small amounts of either block copolymers or end functionalized chains, which segregate, and bond strongly to certain polymer/oxide coated Si interfaces as well as to certain interfaces between this homopolymer and a second, immiscible homopolymer. The amount of such segregation was measured using forward recoil spectrometry and controlled by changing the initial concentration of segregating polymer in the homopolymer layer. He has also discovered that this segregation increases the adhesion of this layer to the substrate. For example a 1μm thick film of polystyrene (PS) formed by spin casting on a silica glass forms an interface which has a fracture energy of approximately 0.3 J/m^2; a similar film suitably doped with a block copolymer of deuterated polystyrene and poly(2-vinylpyridine) (d-PS/PVP) forms an interface which has a fracture energy that is greater than 30 J/m^2. Within the limited funding provided by this grant Kramer has concentrated on quantifying the relationship between the amount of segregated polymer component at the interface and the final volume fraction φ_∞ of segregating polymer in the homopolymer layer well away from the interface. The amount of segregated polymer was determined as the interface excess z^*_i defined as

$$z_i^* = \int_0^\infty dz\,(\varphi(z) - \varphi_\infty) \qquad (1)$$

where z is the distance normal to the interface and $\varphi(z)$ is the volume fraction at that distance. The integral excess is likely to be the most important quantity controlling the fracture properties of the modified interface, since ν_i, the number of segregating chains per unit area at the interface, is directly proportional to it, as given by the following relationship:

$$\nu_i = z_i^* \rho_o / N_c \qquad (2)$$

where ρ_o is the segregating polymer segment density and N_c is the degree of polymerization of that segregating polymer.

One set of experiments measured the segregation of a diblock copolymer of deuterated polystyrene (d-PS) and poly2vinylpyridine (PVP) to silicon covered with its native oxide. The segregation was measured by spin casting a thin film from a solution of PS and a small amount of the d-PS/PVP block copolymer on a silicon wafer with a thin (~20Å) native oxide layer. Usually little if any segregation occurred during the spin casting because of kinetic limitations. However after the film was annealed for a sufficient time above the glass transition temperature T_g of the PS, the block copolymer segregated strongly to the interface. The segregation before and after annealing was measured by forward recoil spectrometry FRES. Figure 1 shows the $\varphi(z)$ profile derived from a FRES spectrum from an annealed PS film, 5000Å thick, which had a bulk volume fraction $\varphi_\infty = 0.003$ of d-PS/PVP block copolymer, the degree of polymerization of whose blocks were 391 for the d-PS and 68 for the PVP. The integral interface excess was found by using a FRES simulation program (RUMP) and matching the integrals of the experimental and simulated interface peaks; in Figure 1 it corresponds to 48Å.

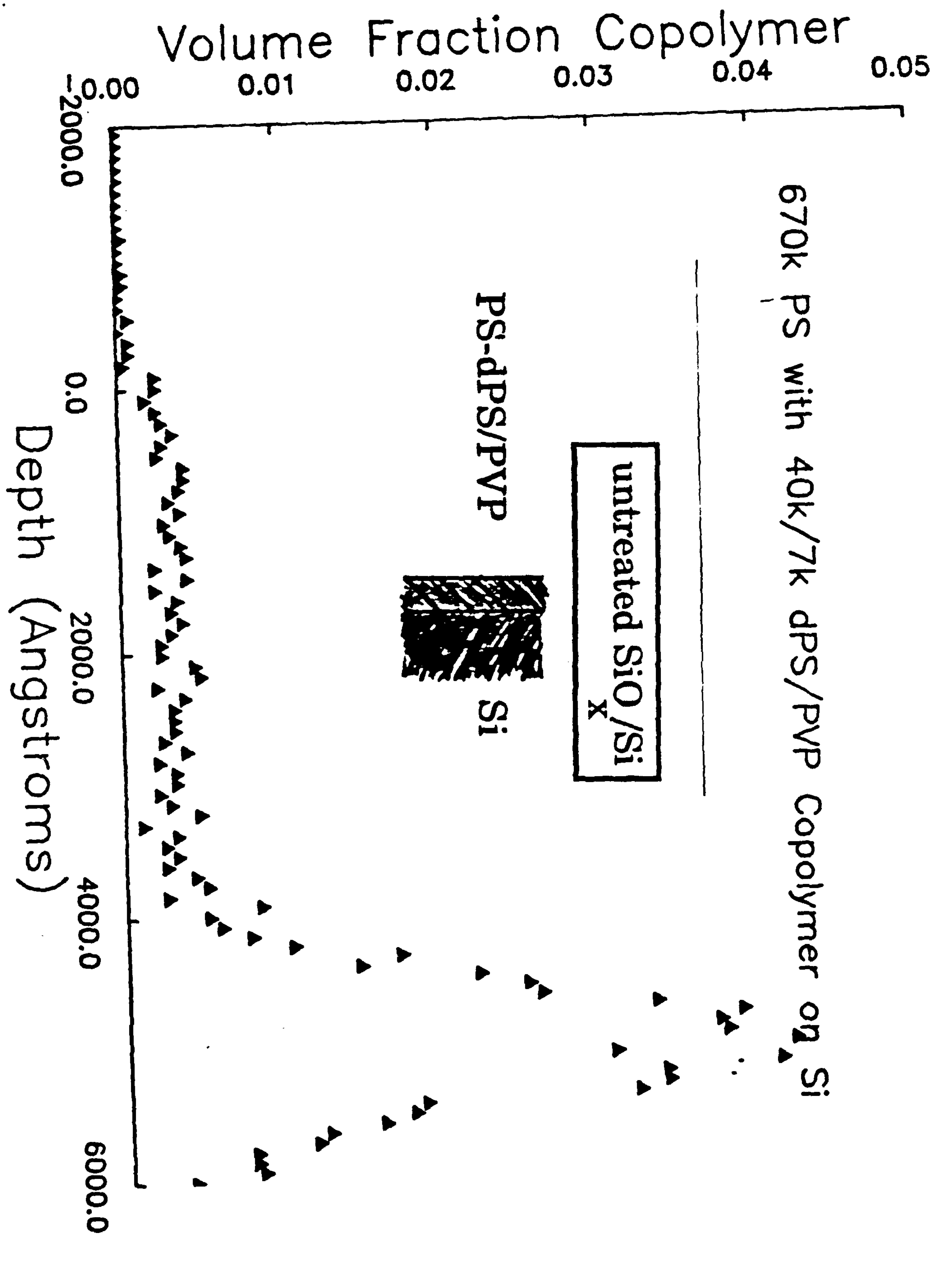
The segregation of deuterated polystyrene (d-PS)/ poly-2-vinylpyridine block copolymers in polystyrene to the interface between this polymer and silicon was measured as a function of φ_∞. If the surface of the silicon is covered with its usual thin native oxide layer, such block copolymers segregate strongly to the interface, with the integral excess being often 75Å or more, as shown in Figure 2 ("untreated" Si). Even if the oxide layer is stripped (by an HF treatment), the block copolymer segregates strongly to the interface. (The fact that the Si interface with the polymer remains free of oxide even after annealing can be verified by dissolving away the polymer layer and measuring the contact angle of the Si surface with water.)

If however the Si surface is reacted with octadecyltrichlorosilane (OTS) prior to spinning on the polymer layer, there is no measurable segregation of the block copolymer to the Si interface at low volume fractions of block copolymer. (The OTS forms a so-called self assembled monolayer of C_{18} paraffin chains 27Å thick on top of the SiO_x.) A depth profile from one such sample is shown in Figure 3. This experiment demonstrates for the first time that the detailed nature of the Si interface is vital in controlling the block copolymer segregation and that relatively small changes in the chemistry of the interface can change the segregation behavior drastically. It is interesting to note that at somewhat higher bulk volume fractions a small amount of segregation to the OTS-coated Si is observed. We tentatively attribute this segregation to long-range forces between the polar PVP and the SiO_x layer under the OTS. If this explanation is correct, the amount of the segregation should be a strong function of the thickness of the paraffin layer, being much weaker for thicker layers. We plan to test this hypothesis using alkylsilanes with alkane block ranging from C_{12} to C_{24} instead of OTS to form the self assembled paraffin monolayer.

If the homopolymer polystyrene is of high molecular weight segregation of the block copolymer at higher nominal block copolymer concentrations is observed at both the free surface of the polymer film and the OTS-treated Si interface. From results of previous experiments on block copolymer segregation at the interface between PS and PVP homopolymers where a similar effect was observed and proved to be due to the segregation of block copolymer micelles, not individual block copolymer chains, this sudden onset of segregation, where none was present at lower concentrations, is thought to be due to a similar micelle segregation. The driving force for this micelle segregation is not the enthalpic binding of the polar PVP to the Si interface as it is in the case of the segregation of individual block polymer chains to the bare Si, but to the fact that the interface between the d-PS chains in the "corona" of the micelle has lower entropy (and thus higher free energy) than the pure homopolymer. The same is true of any abrupt interface with the homopolymer. By combining two such interfaces together, for example the micelle-homopolymer and the Si-homopolymer, the total free energy of the system is decreased.

While the theoretical modelling of the segregation of block copolymer chains to rigid, nonpolymer interfaces like Si is just in its infancy, we expect successful models to be close to the models of the segregation of block copolymer chains to interfaces between the two homopolymers. We have tested such models by measuring the segregation of the same block copolymer of d-PS and PVP (DP 391/68) to interfaces between PVP homopolymer of high molecular weight (600,000) and PS homopolymer whose molecular weight weights were 670,000, 233,000, 127,000 and 9,000. Very little difference in segregation is observed between the different high homopolymer molecular weights as seen in Figure 4, but a significant decrease in segregation is found at the lowest PS molecular weight of 9000. The models of the segregation differ in their treatment of the stretching of block copolymer chains to form a "brush" at the interface and their assumptions about the penetration of the "brush" by the homopolymer. The Leibler model, which assumes that the stretching and the penetration is uniform can approximately reproduce the shape of the segregation isotherm but requires that the Flory segment-segment interaction parameter χ between PS and PVP increase by more than a factor of 2 between the high and low Mw PS samples in order to produce a good fit to the data. In contrast a self-consistent mean field model, which makes no assumption about uniformity of stretching of the brush or the homopolymer penetration of it, can accurately predict the segregation isotherm at all PS molecular weights using a single value of χ. Figures 5a and 5b show the comparison between the data for the various cases and the prediction of the models.

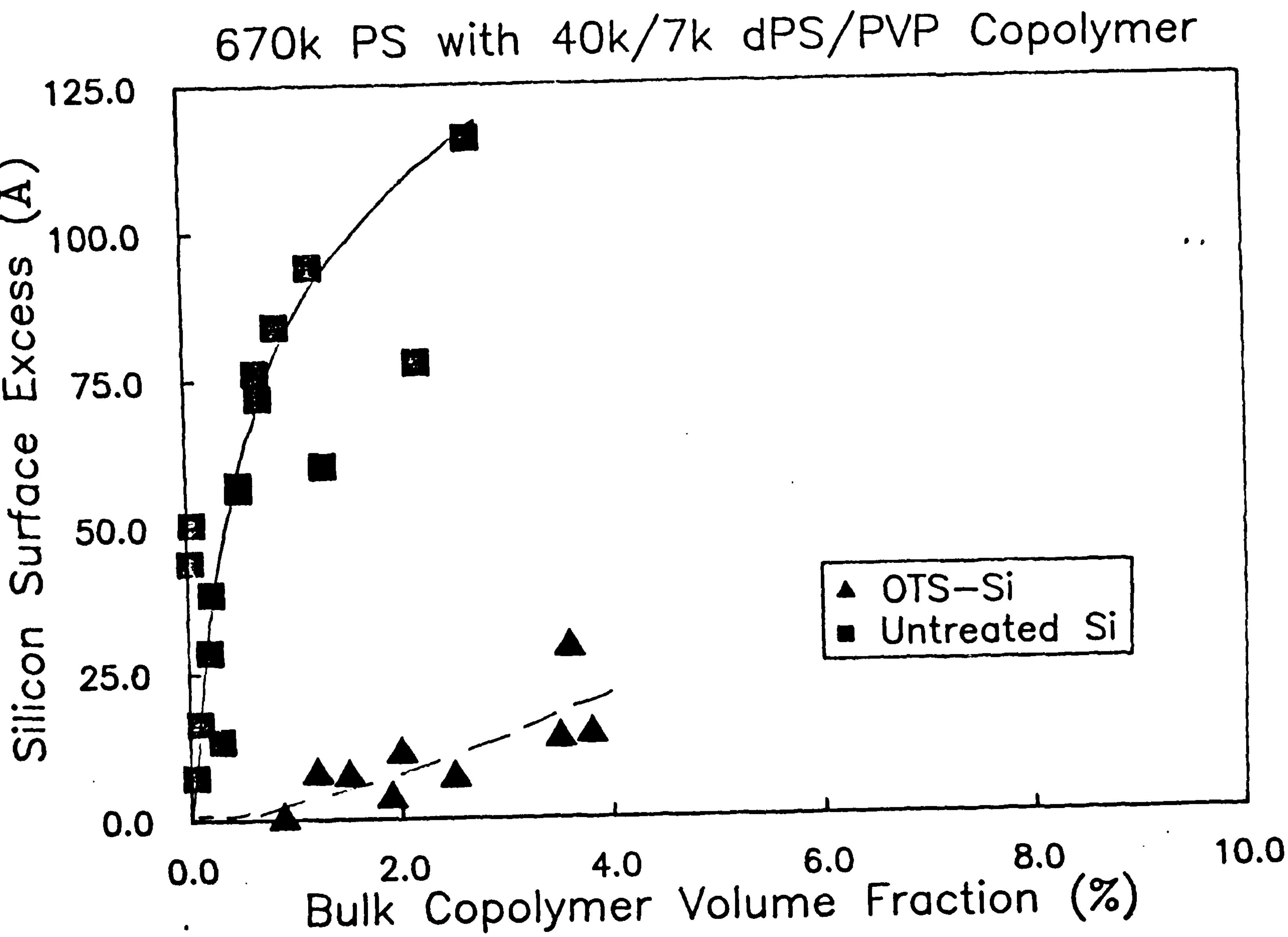
The fundamentals that have been discovered here promise to improve the control of cracking along interfaces between a rigid polymer and a non-polymeric material which is of interest in a number of critical electronic packaging applications.

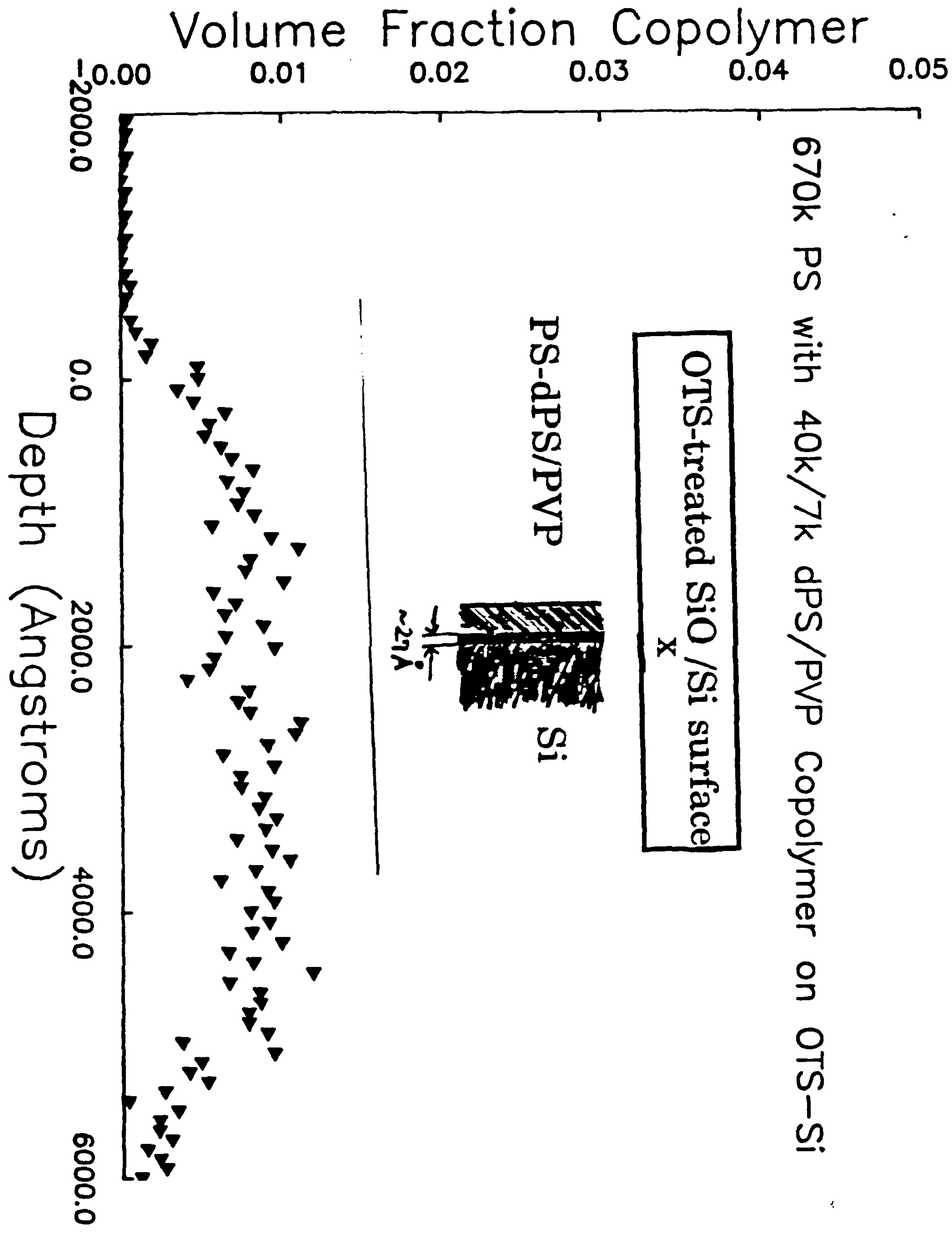


Volume Fraction Copolymer
0.00
0.01
0.02
0.03
0.04
0.05
670k PS with 40k/7k dPS/PVP Copolymer on Si
untreated SiO$_x$/Si
PS-dPS/PVP
Si
-2000.0
0.0
2000.0
4000.0
6000.0
Depth (Angstroms)

FIG. 1

FIG. 2

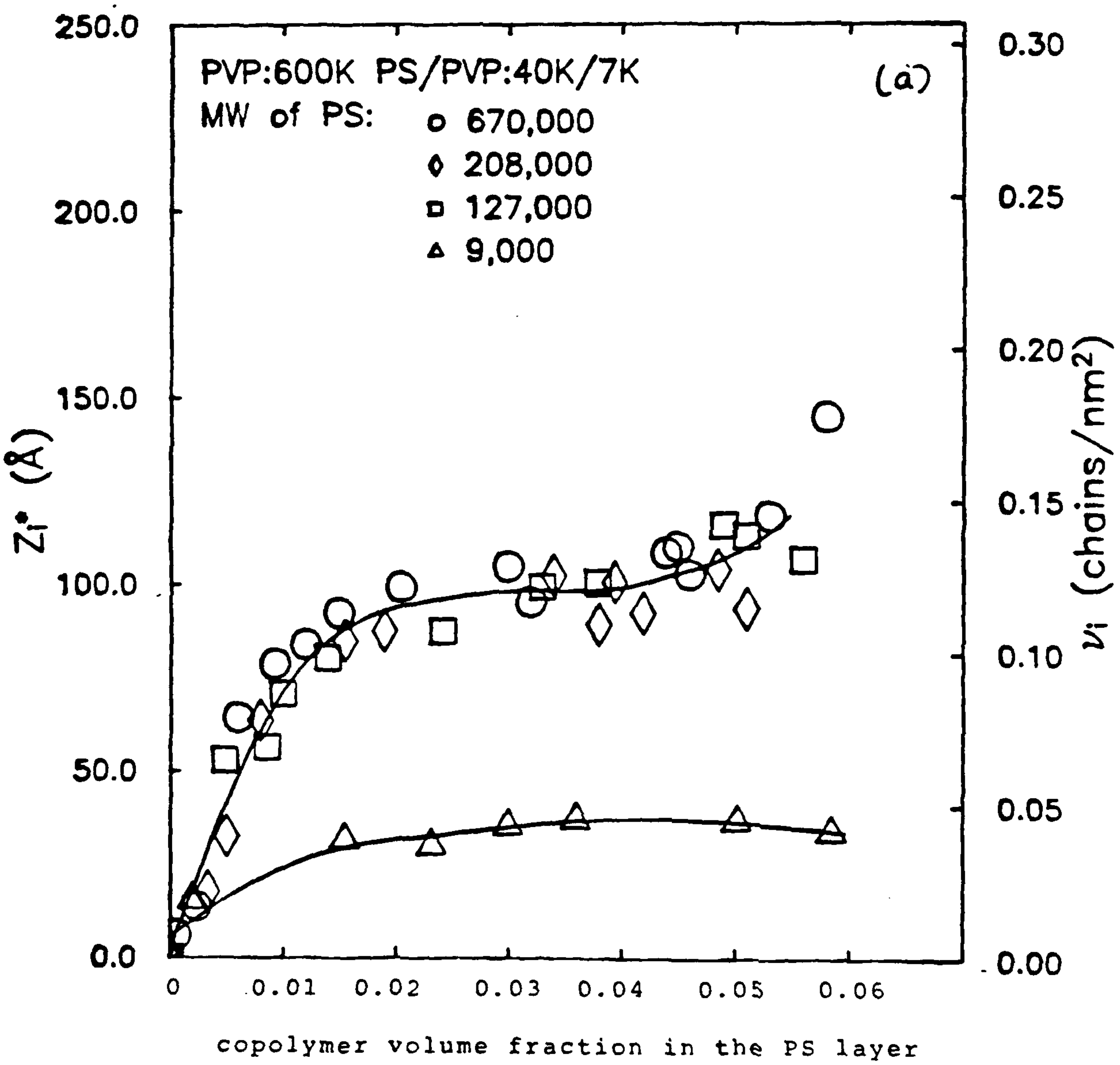

670k PS with 40k/7k dPS/PVP Copolymer
Silicon Surface Excess (Å)
125.0
100.0
75.0
50.0
25.0
0.0
0.0
2.0
4.0
6.0
8.0
10.0
Bulk Copolymer Volume Fraction (%)
OTS–Si
Untreated Si



670k PS with 40k/7k dPS/PVP Copolymer on OTS–Si
Volume Fraction Copolymer
-0.00
0.01
0.02
0.03
0.04
0.05
Depth (Angstroms)
-2000.0
0.0
2000.0
4000.0
6000.0
OTS-treated SiO /Si surface
x
PS-dPS/PVP
Si
~27Å

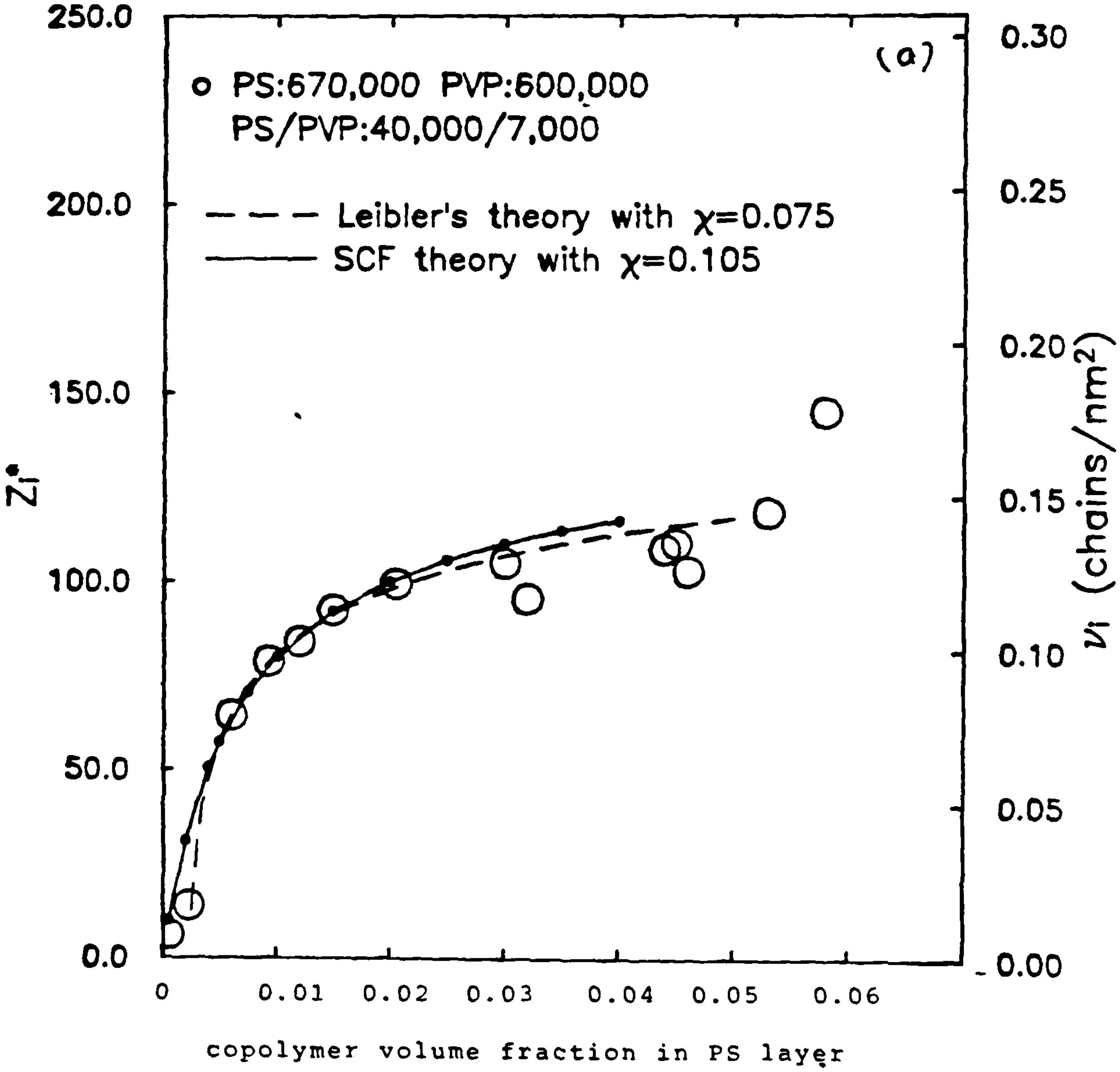
FIG. 3

FIG. 4



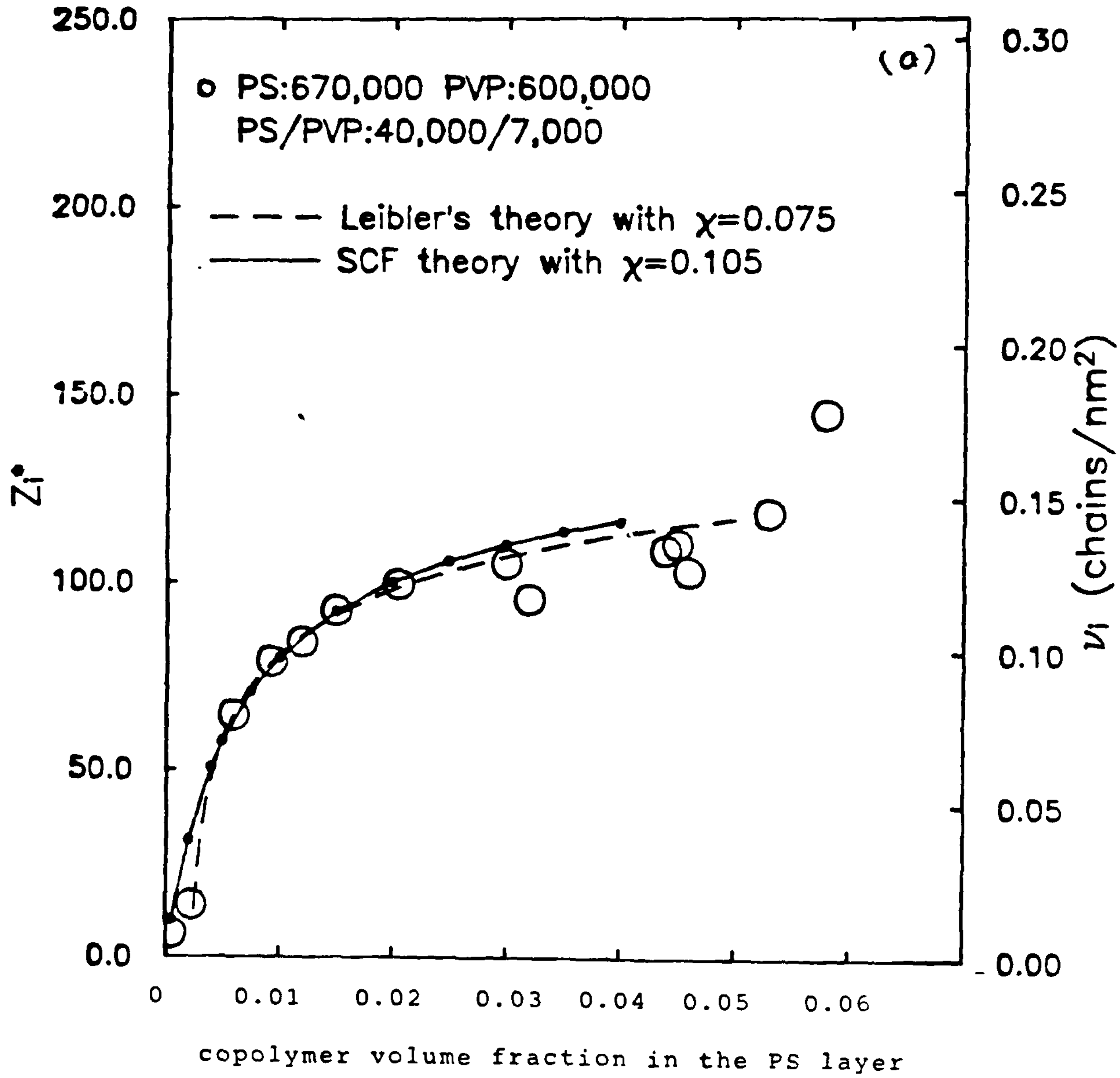
PVP:600K PS/PVP:40K/7K
(a)
MW of PS:
670,000
208,000
127,000
9,000
250.0
200.0
150.0
100.0
50.0
0.0
0.30
0.25
0.20
0.15
0.10
0.05
0.00
Z_i^* (Å)
ν_i (chains/nm^2)
0
0.01
0.02
0.03
0.04
0.05
0.06
copolymer volume fraction in the PS layer

FIG. 5A



250.0
200.0
150.0
100.0
50.0
0.0
Z_I^*
0.30
0.25
0.20
0.15
0.10
0.05
0.00
ν_I (chains/nm^2)
(a)
o PS:670,000 PVP:600,000
PS/PVP:40,000/7,000
Leibler's theory with χ=0.075
SCF theory with χ=0.105
0
0.01
0.02
0.03
0.04
0.05
0.06
copolymer volume fraction in PS layer

FIG. 5B


(a)
o PS:670,000 PVP:600,000
PS/PVP:40,000/7,000
Leibler's theory with χ=0.075
SCF theory with χ=0.105
250.0
200.0
150.0
100.0
50.0
0.0
Z_i^*
0.30
0.25
0.20
0.15
0.10
0.05
0.00
ν_i (chains/nm^2)
0
0.01
0.02
0.03
0.04
0.05
0.06
copolymer volume fraction in the PS layer

RESEARCH SUMMARY: C.-Y. LI

A continuous microindentation test system which is capable of measuring the load and indentation depth response during indentation of submicron films deposited on a substrate has been developed. Such a test system has been used to investigate the variation of the hardness and load relaxation properties of thin films as a function of the depth of indentation. It is shown that the hardness of a thin film deposited on the hard substrate consists of a part which is indentation depth independent and a part of which is dependent on the depth of indentation. The first part is influenced by the presence of residual stresses as well as by the hard substrate acting as a barrier to dislocation motion in the case of a metal film. The latter part is influenced by the contained deformation, as such, is dependent on the level of adhesion between the film and the substrate. It is possible therefore to use the indentation depth dependence of the hardness as a quantitative measure of interfacial adhesion.

The effects of constrained deformation are also manifested in the indentation depth dependent load relaxation properties of the film. The indentation load relaxation properties provide an effective means to probe the local deformation properties of the film. It is found that near the film/substrate interface not only the hardness is significantly increased, the stress dependence of the strain rate is also changed.

These techniques are used to investigate the deformation and adhesion properties of several metal films deposited of oxidized silicon.

RESEARCH SUMMARY: A. L. RUOFF

Adhesion enhancement of polyimide on copper was created by ion beam etching of copper with argon with the result that adhesion strength was doubled.

Adhesion enhancement of copper on polyimide was caused by reactive ion beam etching of polyimide with oxygen with the result that the adhesion strength was increased by a factor of twenty five.

The mechanisms of the enhancement have been proposed. Polymide/Cu system is of current technological interest.

LIST OF PUBLICATIONS UNDER ARO SPONSORSHIP

1. "On the Reaction of Thin Copper Oxide Films with Alumina Substrates", D. W. Susnitzky and C. B. Carter, submitted to J. Materials Science (1990).

2. "Study of Surface Phenomena in Ceramics by TEM", D. W. Susnitzky and C. B. Carter, Materials Science Center, Cornell University, Report No. P002 (1989).

3. "Metal-Ceramic Interphase Interfaces: Preparation and Structural Characterization", D.W. Susnitzky, S. R. Summerfelt and C. B. Carter, Mat. Res. Soc. Symp. Proc., vol. 122, pp. 541-546 (1988).

4. "The Formation of Copper Aluminate by Solid-State Reaction", D. W. Susnitzky and C. B. Carter, Materials Science Center, Cornell University, Report No. P041 (1990), submitted to J. Materials Research.

5. "Direct Observation of Microstructural Changes in Ion-Beam-Modified Ceramics", M. G. Norton, E. L. Fleischer, W. Hertl, J. W. Mayer and C. B. Carter, Materials Science Center, Report No. D012 submitted to proceedings of 12th International Congress on Electron Microscopy, Seattle.

6. "Investigation of the Adhesion of Chromium on Sol-Modified Ceramic Surfaces", DeMott, G. J. Ph.d. Thesis, Cornell University, 1989.

7. Burlitch, J. M., DeMott, G. J., and D. L. Kohlstedt, Mat. Res. Soc. Sym. Proc. on Chemical Perspectives of Microelectronic Materials, 131, pp. 454-458, 1989.

8. "Adhesion of Chromium Metallization on Alumina Surfaces Prepared by Sol-Gel Techniques", Kanai, H., G. J. DeMott, and D. L. Kohlstedt, J. Mat. Sci., in press (1990)

9. "Hardness and Adhesion Measurements of Copper Metallizations by a Continuous Indentation Approach" W. R. LaFontaine and Che-Yu Li, to be published in the proceedings of Symposium on Electronic Packaging Materials Science, MRS meeting, winter,1990.

10. "Effect of Residual Stress and Adhesion on the Hardness of Copper Films Deposited on Silicon", W. R. LaFontaine, B. Yost, and Che-Yu Li, J. Mater. Res., Vol. 5, No. 4, April 1990.

11. "Indentation and Load Relaxation Experiments with Indentation Depth in the Submicron Range", W. R. LaFontaine, B. Yost, R. D. Black, and C-Y Li, J. Mater. Res., Vol. 5, No. 10, Oct. 1990.

12. "Development of new ion beam modification techniques to enhance copper and polyimide (PI) adhesion in multilevel electronic packaging", Kyung W. Paik and Arthur L. Ruoff Mat. Res. Soc. Symp. Proc. 154, 21 (1989).

13. "Adhesion enhancement of thin copper film as polyimide modified by oxygen reactive ion etching", Kyung W. Paik and Arthur L. Ruoff J. Adhesion Science & Technology, 4, 465 (1990).

LIST OF SCIENTIFIC PERSONNEL EMPLOYED ON THIS PROJECT

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DeMott, G. J - Graduate Student. Received Ph.d 1989.

LaFontaine, W. R. - Graduate Student. Received Ph.D 1990.

McKernan, S. - Postdoc. Partial support.

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Simpson, Y. K. - Graduate Student, received Ph.D in 1988.

Susnitzky, D. W. - Graduate Student, received Ph.D in 1988.

Carter, C. B. - Professor.

Kramer, E. J. - Professor.

Kohlstedt D. L. - Professor.

Li, C.-Y. - Professor.

Ruoff, A. L. - Professor.